UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
NATIONAL EXPOSURE RESEARCH LABORATORY
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OFFICE OF
RESEARCH AND DEVELOPMENT

MEMORANDUM

SUBJECT: Perchlorate Interim Technical Report

FROM: Ken W. Brown, Director, TSC
Environmental Sciences Division

TO: Kevin Mayer, Acting Associate Chief (SFD-7)
Region IX

Kevin, please find attached the report titled "Interim Technical Report Methods and Quality Assurance for the Analysis of Perchlorate in Water" dated November 12, 1997. As you will note, the CAL-DHS method appears to be satisfactory as a function of the literature survey assessing available laboratory data and the results of some limited tests that the TSC has conducted thus far.

Included in the attached report are recommendations for further studies. The TSC is planning to investigate the identified studies as I feel the results will provide significant input in assessing data quality.

I have taken the liberty of sending Brenda Pohlmann a copy of this report. As you are aware Brenda is the Remedial Action Program Supervisor for the Nevada State Department of Conservation and Natural Resources, Department of Environmental Protection.

cc: with Attachment
Brenda Pohlmann, State of Nevada

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12 NOV. 1997

Interim Technical Report
Methods and Quality Assurance for the Analysis of Perchlorate in Water

Superfund Technical Support Product
in support of EPA Region 9
San Francisco, CA

J. R. Donnelly
Lockheed Martin Environmental Services

November, 1997

Report Prepared for:
U.S. Environmental Protection Agency
Technology Support Center for
Monitoring and Site Characterization
National Exposure Research Laboratory
Environmental Sciences Division
Las Vegas, Nevada 89193-3478

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I. INTRODUCTION

The Region 9 Superfund site Cleanup Branch Chief requested that the EPA-Las Vegas Technical Support Center critically review the California Department of Health Services (DHS) analytical protocol for the detection of perchlorate to the 4 ppb reporting limit. This ion chromatographic (IC) method has been used to detect perchlorate in groundwater and surface water supplies in California, Arizona, and Nevada. It is considered important to establish the reliability of this method to detect perchlorate to the 1 ppb estimated detection limit in support of the California level of concern that is set at 18 ppb. Additionally, effects of high dissolved solids should be addressed because of the presence of high dissolved solids typically present in groundwater and surface water in the arid southwest.

This report discusses studies that were made as part of an assessment of Cal-DHS method, the identification of potential alternative methodology, and studies that have been initiated to answer questions about sample holding times and stability. Recommendations for future work are included, with the emphasis on determining the level of data quality that can be expected for routine monitoring by the Cal-DHS method.

II. OBJECTIVES AND APPROACH

The California DHS method was provided, and some data were available so that an assessment of method capabilities could be made. The capabilities of the method addressed by this report and by planned studies include the following: false positives, false negatives, intra-laboratory and inter-laboratory precision and accuracy, sample holding times, sample preservation, and matrix effects such as dissolved solids/conductivity.

To address the request from Region 9, a search of the chemical literature was initiated using the chemical substance indices of Chemical Abstracts. The indices were checked from 1967 through 1997, and the abstracts of potentially relevant publications and patents were reviewed. Based upon these reviews, potentially relevant complete articles were obtained and reviewed as well. Perchlorate, ammonium perchlorate, sodium perchlorate, and potassium perchlorate were searched. The reason for searching the latter two salts is a combination of the fact that these cations are present in relatively high concentrations in water systems, and that sodium and potassium perchlorate salts have been subjected to considerable research having potential relevance. Once the ammonium perchlorate enters the water system, the ammonium cation usually becomes insignificant.

The American Water Works Association Research Foundation (AWWARF) workshop for the Perchlorate Research Issue Group was

attended on September 30--October 2, 1997. At this workshop, participants separated into workgroups matching their different specialties and interests in order to develop the research needs of the specialties into specific plans. These specialties/interests included Analytical, Utility/Regulatory, and Treatment Options. Four areas for possible future work on analytical methodology were identified. Among the participants in the analytical workgroup were Kevin Mayer, EPA Region 9, and Howard Okamoto, California-DHS. These research areas were considered valuable unanimously, and the texts were reviewed and agreed upon by all participants of the analytical workgroup. Summaries of these plans are included as Appendix A.

III. PRELIMINARY FINDINGS

The literature search located potentially relevant publications from 1967 to present. Several research type analytical methods were found in the scientific literature. Ion chromatography (IC), such as that used in the DHS method, has been employed for inorganic anions including perchlorate. Therefore, the basic technology used in the DHS method is reasonable. Most research methods have been intended to detect ppm concentration levels or above, and the DHS reported 1 ppb detection limit can be considered state-of-the-art for an analytical method with routine sample determination as its goal.

The potential for false positives and false negatives is worth studying. Research endeavors have provided outlines for such investigations using capillary electrophoresis (CE) and IC separations with a variety of detection systems including ion spray and electrospray mass spectrometry, conductivity, and Raman spectroscopy. Complementary analysis by one of these techniques would address and resolve interference issues regarding the confirmatory power of an IC method.

Dionex Corporation (Sunnyvale, CA) has recently developed an alternative IC method for perchlorates. Limited documentation on this method is available; it is included as Appendix B. This method primarily involves the use of a different ion chromatographic column without the use of p-cyanophenol. This method has the potential to improve the confirmatory power of IC because a different column with somewhat different retention properties is used. Within the context of environmental monitoring methods, it is potentially useful as a "second column confirmation" type of technique, somewhat analogous to the use of two different-polarity gas chromatographic columns for organic analysis.

No data were found in the chemical literature on perchlorate sample holding times and preservation. Some data are available on chlorite and chlorate. The latter is reportedly stable for 18

days without a preservative. Reportedly, perchlorate formation and decomposition are unfavorable kinetically in such chemical systems, so the target anion should be much more stable than chlorate. In support of this hypothesis, LAS laboratory personnel stated that they have observed no degradation of perchlorate standards over time. We recommend conducting a sample holding time study, using low level environmental samples. We may be able to access a set of samples initially analyzed about three months ago. Three of these samples represent a range of conductivities: 10 ppb, 955 μS/cm; 47 ppb, 1830 μS/cm; and 14 ppb, 6780 μS/cm (see the Recommended Further Studies section of this report for details).

The thermodynamics of perchlorate decomposition are favorable. Thus, it is potentially a powerful oxidizer. The kinetics are slow at ambient temperatures and in the absence of catalysis. It is likely that a number of biological systems provide biochemical catalysis of the decomposition process, because several biodegradation schemes have been developed for environmental remediation. However, it is not clear whether any such biological systems are likely to be present in drinking water sources. If not, then the perchlorate anion would be expected to have great stability in the surface or ground water.

Limited data (for one sample set) were available from two laboratories. The results for low concentration samples (6 to 47 ppb) often varied up to about a factor of two between the laboratories. Higher level (440 to 1620 ppb) sample reported concentrations varied less than 20%. One laboratory reported results consistently higher than the other laboratory, suggesting that a difference may exist in actual vs. nominal concentrations of the perchlorate standards at one or both laboratories. Overall, these analytical differences are reasonable for low and high concentration inter-laboratory results. Differences in standards could account for much of the difference. The frequent use of inter-laboratory check samples could help to identify such problems or differences with analytical standards.

Sample conductivities in that set were measured to be 955 to 6780 μS/cm. Conductivity was apparently not a factor in inter-laboratory data variability. LAS Laboratories reported testing the DHS method up to 7000 μS/cm, with satisfactory results. However, it is expected that suppression will be encountered with conductivities that are over 10,000 μS/cm.

Field samples are typically filtered with an Acro-Disc before analysis. Using a 25 ppb standard, it was found that this filter does not adsorb perchlorate. Filtered and unfiltered standards gave the same analytical results.

The literature search, available analytical data from two laboratories, and experimental checks of (a) the filtering step,

(b) method performance at conductivities up to 7000 μS/cm, and (c) the stability of standards over time all indicate that the Cal-DHS method is conditionally satisfactory within the test limits. In Section IV, further studies are outlined to validate the method and to determine its level of performance more completely. It should be noted that the method should be tested at higher conductivities, holding times need to be established, positive identity of the perchlorate peak needs to be made, and freedom of the perchlorate peak from analytical interferences needs to be verified.

IV. RECOMMENDED FURTHER STUDIES

Four analytical studies were proposed in the AWWARF Perchlorate Issue Group workshop (see Appendix A). These include (a) the single-laboratory optimization of certain method parameters, including verification that the peak identified as perchlorate is correctly identified and is interference free, (b) a survey of existing data from prior analytical work to assess the performance of the Cal-DHS method, (c) the development of an alternative method suitable for routine analysis, and (d) an inter-laboratory study to determine the performance of the Cal-DHS method.

Several relatively straightforward laboratory-related issues are recommended for initial study in support of part (a), above. These studies are described below, and are intended to address issues regarding sample holding time, analyte adsorption to container walls, sample preservation, and sample storage.

Sample holding time study

This study would be designed to test sample holding times and perchlorate stability in water samples and standard solutions. All samples and standards should be analyzed in triplicate. The field samples selected for this holding time study should include low concentrations above and below the interim action level of 18 ppb, and different commonly-encountered conductivities. Lower concentrations should be selected so that even small changes in concentration due to adsorption would be noticeable, and to tell whether long holding times might reduce the concentrations of perchlorate in low level samples to below the action level, or even to non-detected levels. For this study, holding times should begin with periods as short as one or two weeks. Preliminary findings indicate that perchlorate field samples may degrade even under refrigeration, with half lives of perhaps 3 months.

Analyte adsorption study

One mode of perchlorate loss could be adsorption onto the walls of the container. An aliquot (~25mL) of each of the above-described sample types (10-50 ppb, low, medium, and high conductivity) should be dispensed into each type of container commonly used: cleaned borosilicate (Pyrex;™ typically used for analytical standards), soda lime glass (bottles used for field samples), and high density polyethylene plastic containers (HDPE, frequently used in inorganic chemistry sampling and analysis). One of each sample/container type should be stored at room temperature, and one under refrigeration. This study should be conducted along with the holding time study, so that adsorption characteristics (possibly dependent upon material type) and degradation phenomena (possibly dependent upon time, temperature, etc.) can be assessed together.

Conductivity study

The range of sample conductivity where IC response is linear to perchlorate concentration should be determined. At higher conductivities than about 10,000 μS/cm, suppression may occur, rendering the response non-linear. It should be determined whether this non-linearity can be reliably quantified and whether adequate response vs. concentration calibrations can be performed at high sample conductivities. It is not presently known whether high conductivity samples can be reliably quantitated.

APPENDIX A

SUMMARIES OF RECOMMENDED RESEARCH PROJECTS

FROM THE AWWARF PERCHLORATE ISSUE GROUP WORKSHOP

TITLE: SHORT-TERM PERCHLORATE LABORATORY ISSUES

Description of Problem: The California Department of Health Services (Cal-DHS) recently developed a new, draft method for the analysis of perchlorate in water matrices. The method is based upon ion chromatography with conductivity detection. This method has a reported quantification limit of 4 PPB. Although this method appears to meet scientific requirements, some Laboratory-based studies are needed to answer Cal-DHS method performance questions. The answers are needed to finalize the draft method and to validate it for regulatory purposes.

Objective of Research Response: Develop an "interim method" with expanded QA/QC for the purpose of wide spread distribution and future inter-laboratory study

Recommended Funding: $100K

Past and Ongoing Related Research: Cal-DHS has some method performance data already, but critical questions remain to be answered.

Proposal Source: Perchlorate Issue Group

Controversial Issues:

Audience: Check all high priority audiences, circle the highest priority.

☒ **Utilities**	☒ Regulators	☐ Research Community	☐ Consultant
☐ AWWA	☐ Other Orgs.	☐ Collaborative Partners	☐ Manufacturers
☒ Congress	☐ Education	☐ Environmental Groups	☐ AWWARF (internal)

Utility Audience: Check one.

☐ Manager	☒ Water quality	☐ Design and engineering	☐ Administration
☐ Public affairs	☐ Operations	☐ Other_Customers_____	

Position on Research Continuum: Check one.

☐ Proof of concept	☐ Develop application	☐ Laboratory test	☐ Pilot test
☐ Full-scale test	☒ Implementation barrier	☐ Product development	

BACKGROUND

The experiments will provide answers about method features and its level of performance in support of regulatory and utility users.

RESEARCH APPROACH

Prepare an experimental design, conduct lab tests, and assess results. The results of this work will be submitted in a final report. Topics of this study include:

- determine separations achieved from the perchlorate peak by potentially interfering anions;
- determine appropriate sample holding time limits, degree of adsorption of perchlorate on surfaces of vessels and equipment, need for sample preservation, and procedures for sample storage;
- investigate sample cleanup and pre-concentration procedures to improve detection limits and method reliability on environmental aqueous matrices;
- verify the effectiveness of dilution, spiking, and fortification procedures that are being used for difficult samples and for verification of method accuracy;
- verify that the method is perchlorate-specific for field samples by developing a research-grade procedure for validating results obtained by the primary Cal-DHS method.

TITLE: SURVEY THE PERFORMANCE OF THE CALIFORNIA DHS (ION CHROMATOGRAPHY) ANALYTICAL PROTOCOL

Description of Problem: Important water supply decisions are being made on the basis of perchlorate analytical results from laboratories following the California Department of Health Services (Cal-DHS) ion chromatography analytical protocol. Numerous inter-lab and intra-lab QA procedures have been performed, but have not been gathered into a single document for evaluation of the methodology.

Objective of Research Response: This research will bring together all available information on the performance of the Cal-DHS perchlorate and evaluate the method for analysis of low concentrations of perchlorate in surface water and groundwater samples. The document will be used for formal method approval as well as further method development.

Recommended funding: $75,000

Past and Ongoing Related Research: California DHS Method development, Cal DHS lab certification program, USEPA and US Air Force method evaluation studies, various inter-lab and intra-lab QA/QC results.

Proposal Source: Perchlorate Issue Group

Controversial Issues: The method must be acceptable to EPA, state agencies, and Standard Methods.

Staff Comments:

Audience: Check all high priority audiences, circle the highest priority.

☒ **Utilities**	☒ Regulators	☒ Research Community	☐ Consultant
☐ AWWA	☐ Other Orgs.	☐ Collaborative Partners	☐ Manufacturers
☐ Congress	☐ Education	☐ Environmental Groups	☐ AWWARF (internal)

Utility Audience: Check one.

☐ Manager	☒ Water quality	☐ Design and engineering	☐ Administration
☐ Public affairs	☐ Operations	☐ Other_____	

Position on Research Continuum: Check one.

☒ Proof of concept	☐ Develop application	☒ Laboratory test	☐ Pilot test
☐ Full-scale test	☐ Implementation barrier	☐ Product development	

BACKGROUND

California DHS developed an enhanced Ion Chromatographic method for the analysis of low concentrations (4 ppb) in February, 1997. Since then at least 6 commercial laboratories in California, four public agency labs and several private and commercial labs outside of California have developed this analytical capability. Several evaluations of the method and performance of various laboratories have been sponsored by US EPA, USAF, Cal DHS,

Nevada Division of Environmental Protection, Southern California Metropolitan Water District, Southern Nevada Water Authority and others. The available performance information needs to be brought together in a single informative document.

Eventual publication of an official analytical protocol as well as widespread acceptance of the method requires an thorough evaluation of the method's performance on a wide range of environmental and water supply samples.

RESEARCH APPROACH

This project is a short-term, high priority study of what is already known regarding the analysis of perchlorate in environmental media. This project will involve gathering available laboratory performance data (inter-lab and intra-lab QA/QC) into a single document. These data, and the lessons learned relative to analysis for perchlorate, will be summarized in the final document. This final document will prove useful in evaluating information pertinent to performance of the method, identifying apparent difficulties due to factors such as water quality, possible interferences and perchlorate concentration.

The initial steps for information gathering relative to this project will involve contacting the California DHS, US EPA, USAF, Cal DHS, Nevada Division of Environmental Protection, Southern California Metropolitan Water District, Southern Nevada Water Authority, California-certified laboratories, Kerr-McGee, Montgomery-Watson and other non-California labs for helpful and useful method information (i.e.: clean-up procedures, matrix effects, etc.).

Laboratory performance data will include results of split and duplicate samples, detection limits on field samples, method modifications or clarifications developed by the laboratories, results on performance evaluation samples or other multi-laboratory determinations, effects introduced by holding time or matrix differences, possible correlations of conductivity, nitrate, sulfate, surfactants, or other anions on method performance.

Project Description author: Kevin Mayer

TITLE: DEVELOPMENT OF AN ANALYTICAL METHOD FOR MEASURING PERCHLORATE ION AT THE 4 PPB LEVEL

Description of Problem: A single ion chromatography method exists for perchlorate measurement. A second method capable of measuring at the 4 ppb level is required for verifying low level concentrations in field samples.

Objective of the Research Response: Develop a low level perchlorate method (4 ppb or lower) applicable for groundwater and surface water field samples that is easily transferable to a water utility. This procedure will be used to verify the existence and concentration of perchlorate in environmental samples.

Recommended Funding: $125,000

Past and Ongoing Related Research: Some CA DHS, other governmental agencies, and numerous private sector corporations (i.e., Kerr-McGee, MWD, Montgomery-Watson).

Proposal Source: Perchlorate Research Issue Group

Controversial Issues:

Staff Comments:

Audience: Check all high priority audiences, circle the highest priority.

☒ **Utilities**	☒ Regulators	☒ Research Community	☐ Consultant
☐ AWWA	☐ Other Orgs.	☐ Collaborative Partners	☐ Manufacturers
☐ Congress	☐ Education	☐ Environmental Groups	☐ AWWARF (internal)

Utility Audience: Check one.

☐ Manager	☒ Water quality	☐ Design and engineering	☐ Administration
☐ Public affairs	☐ Operations	☐ Other_____	

Position on Research Continuum: Check one.

☐ Proof of concept	☐ Develop application	☒ Laboratory test	☐ Pilot test
☐ Full-scale test	☐ Implementation barrier	☐ Product development	

BACKGROUND

Perchlorate has been detected in groundwater and surface waster. The source is believed to be related to the manufacture and use of solid rocket propellants. The USEPA (and the CA DHS) has set a preliminary action level at 4 - 18 ppb of perchlorate. The current ion chromatography method has the capability of measuring 4 ppb. Because water quality in which that perchlorate can be found differs, a second analytical method is sought to verify concentration of field samples.

RESEARCH APPROACH

The developed method is required to measure at the 4 ppb level. The precision at the 4 ppb level should be ± 20%. QA/QC documentation as specific for USEPA acceptable methods are required.

The method to be developed should be based on a different principle than IC or a selective detection method to analyze perchlorate at low concentrations. However, the method should be robust, and should be easily transferable to a water utility laboratory. The types of methods that might be considered, but are not limited to, include:

- capillary electrophoresis
- bioassay methods
- amperometric detectors
- selective reagent methods

The project should start with a comprehensive literature review on the proposed analytical methods and existing understanding of perchlorate chemistry. It is anticipated that a minimum of 4 potential methods should be screened in this project. Further screening should be conducted with a matrix of requirements to rank the potential analytical methods with respect to likelihood of success. Two or 3 analytical methods most likely of success should be pursued for detailed development. The final report should propose detailed procedures for the recommended method.

Project Description author: Bernie Bubnis

TITLE: INTER-LABORATORY STUDY FOR THE PERFORMANCE EVALUATION OF THE CALIFORNIA DHS METHOD: DETERMINATION OF PERCHLORATE BY IC

Description of Problem: A method for the determination of perchlorate by ion chromatography has been recently developed. However, the inter-laboratory accuracy and precision of the method has not yet been determined. This method performance will be critical for regulatory decisions that will need to be made for perchlorate detected near and at the level of concern.

Objective of Research Response: Conduct an inter-laboratory performance evaluation study of the method for the determination of perchlorate by ion chromatography. The study should evaluate the inter-laboratory accuracy and precision of the method.

Recommended Funding: $100,000.

Past and Ongoing Related Research: Limited inter-laboratory date is available. The CA DHS has conducted a limited performance evaluation study for labs in CA performing the analysis for perchlorate using the CA DHS method.

Proposal Source: Perchlorate Issue Group

Controversial Issues: None

Staff Comments: List any pros and cons in support of or against this idea.

Audience: Check all high priority audiences, circle the highest priority.

☒ **Utilities**	☒ Regulators	☒ Research Community	☒ Consultant
☐ AWWA	☐ Other Orgs.	☐ Collaborative Partners	☐ Manufacturers
☐ Congress	☐ Education	☒ Environmental Groups	☐ AWWARF (internal)

Utility Audience: Check one.

☐ Manager	☒ Water quality	☐ Design and engineering	☐ Administration
☐ Public affairs	☐ Operations	☐ Other_____	

Position on Research Continuum: Check one.

☐ Proof of concept	☐ Develop application	☒ Laboratory test	☐ Pilot test
☐ Full-scale test	☐ Implementation barrier	☐ Product development	

BACKGROUND

The California Department of Health Services (DHS) method for perchlorate analysis by ion chromatography has been available since June 1997. Water utilities that are vulnerable to perchlorate contamination have a critical need to monitor their water supplies for this compound.

Therefore, the California DHS method has seen wide-spread use. However, very limited data are available on inter-laboratory performance or the accuracy and precision of this method.

RESEARCH APPROACH

Researchers must be experienced in conducting inter-laboratory performance evaluation studies of laboratory analytical methods. The contractor will supply all performance evaluation samples and will perform the statistical evaluation of the study results. The study should include a variety of water matrices and perchlorate concentration ranges from the quantitation level, regulatory level of concern, and ten times the regulatory level of concern.

Project Description Author: Howard Okamoto

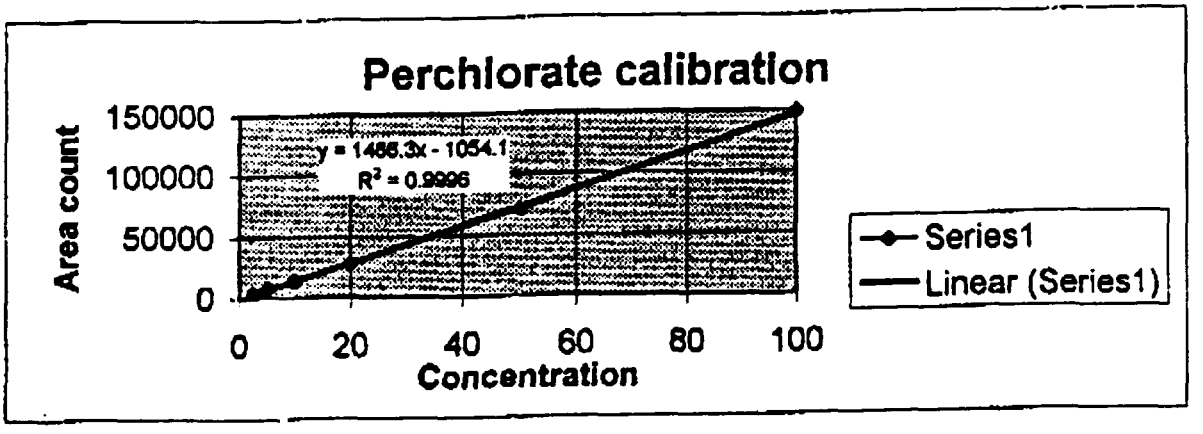
APPENDIX B

DIONEX METHOD FOR PERCHLORATES

DETERMINATION OF PERCHLORATE IN A TAP WATER

Perchlorate calibration

Concnentration(ppb)	Area Coun	Area Count	Average	Response		Average
	1 injection	2 injection				1416.833
2.5	3504	3503	3503.5	1401.4		
5	6818	7018	6918	1383.6		SD
10	14112	13927	14019.5	1401.95		36.48858
20	28025	28141	28083	1404.15		RSD
50	71013	71299	71156	1423.12		2.57
100	148774	148582	148678	1486.78		



Column: IonPac AG11, AS11
Eluent: 100 mM Sodium hydroxide
Flow Rate: 1 mL/min
Inj. Volume: 1000mL
Detection: Suppressed Conductivity, ASRS-II
Autosuppression, external water mode

RSD AND MDL STUDY OF PERCHLORATE

Injection #	Area	Retention time (min)
1	3391	9.48
2	3405	9.57
3	3504	9.50
4	3503	9.45
5	3435	9.47
6	3301	9.52
7	3315	9.43
Average	3407.7	9.49
STD	80.99	0.047
RSD	2.38	0.49

MDL=254 ppt
MDL=SD*$t_{n,99}$ where $t_{n,99}$=3.14 for n=7

Column: IonPac® AG11, AS11
Eluent: 100 mM Sodium hydroxide
Flow Rate: 1 mL/min
Inj. Volume: 1000µL
Detection: Suppressed Conductivity, ASRS-II Autosuppression™, external water mode
2.5 ppb standard injection

Data File :	PEAKNET\DATA\MARK\10304002.DXD	Report Date:	10/30/97 1:11:38 PM
Sample Name:	10PPB Perchlorate	Collected :	10/30/97 11:14:29 AM
Inject # :	2	Vial # :	
Method File:	c:\peaknet\method\mark.met	Calibrated :	Time Unknown
System Name:	Mark	Detector :	CD20
Column Type:	AG11,AS11	Operator :	Mark Laikhtman
Data Points:	3600	Rate :	5.00 Hz
Module Name:	ID:00 01 f4	Moduleware :	1.16

Calibration	Volume	Dilution	Start	Stop	Area Reject	Pk. Width	Threshold
External	1	1	0.00	12.00	1000	10.00	0.50

Filter type: Savitsky-Golay Size:19 points (3.8 sec) 1 iteration(s)

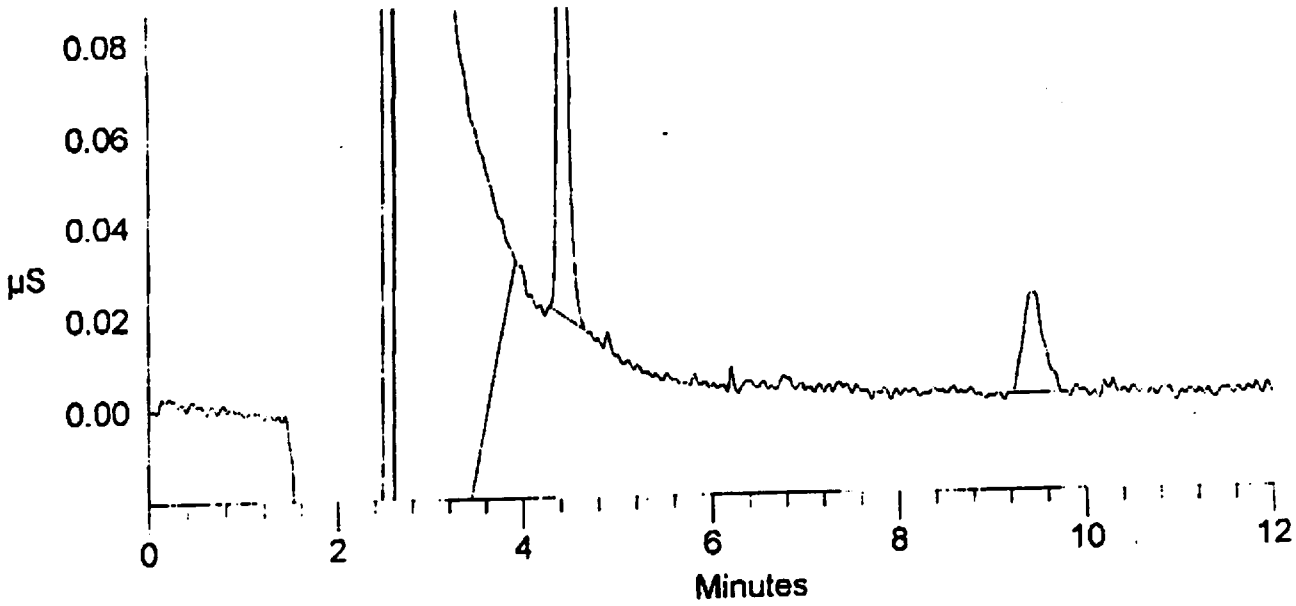
********** Component Report: All Components **********

Pk. Num	Ret Time	Component Name	Concentration	Height	Area	Bl. Code	%Delta
		Totals	0.000	0	0		

********** Peak Report: Unknown Peaks **********

Pk. Num	Ret Time	Component Name	Concentration	Height	Area	Bl. Code	%Delta
1	0.18		0.000	738	7699	1	
2	2.52		0.000	28259	141981	2	
3	2.67		0.000	80161	930667	3	
4	3.35		0.000	4169	37285	4	
5	4.43		0.000	1885	12107	1	
6	9.52		0.000	872	14112	1	
		Totals	0.000	116085	1143852		

File: 10308004.DXD Sample 2.5PPB Perchlorate



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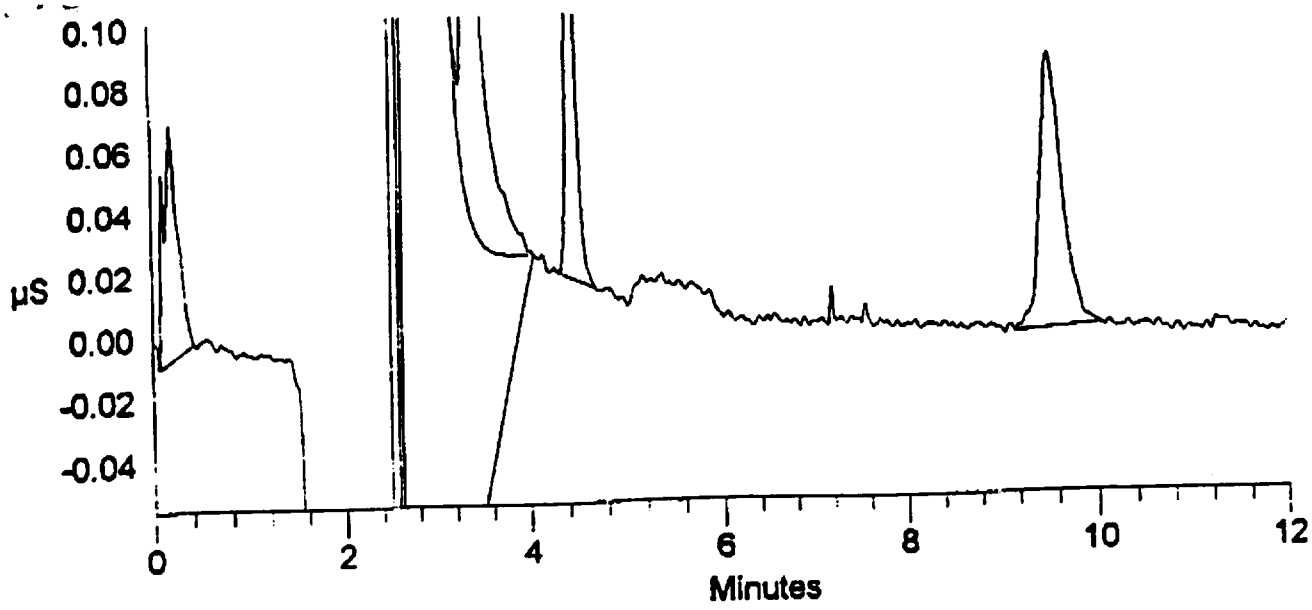
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